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MICROSTRUCTURAL ORIGINS OF HOT SPOTS IN RDX EXPLOSIVE AND A REFERENCE INERT MATERIAL

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W. L. ELBAN, J. C. HOFFSOMMER, AND C. S. COFFEY
RESEARCH AND TECHNOLOGY DEPARTMENT

AND

K. - C. YOO* AND R. G. ROSEMEIER*

*MECHANICAL ENGINEERING DEPARTMENT
UNIVERSITY OF MARYLAND, COLLEGE PARK

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PREPARED FOR
OFFICE OF NAVAL RESEARCH
800 N. QUINCY STREET
ARLINGTON, VA 22217

NAVAL SURFACE WEAPONS CENTER

Dahlgren, Virginia 22448 • Silver Spring, Maryland 20910

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Hot Spot	Laue X-ray Diffraction	Chemical Analysis
RDX Explosive	Hardness	Fractoemission
MgF_2	Microhardness	Ammonium Perchlorate
Berg-Barrett X-ray Topography	Drop-Weight Testing	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The crystalline perfection in production-grade RDX material has been investigated using Berg-Barrett and Laue x-ray diffraction techniques. Particularly noteworthy is the observation by x-ray topography that pores within the crystals appear to be surrounded by a strain-free matrix. Fracto-emission experiments on production-grade and NSWC laboratory-grown RDX were performed in collaboration with Washington State University researchers. The purpose was to study the early stages of decomposition resulting from fracture. Different electron emission behaviors were observed and attributed

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to the nature of the fracture that occurred. Gas chromatographic analyses were performed on production-grade RDX samples that had been impact loaded to investigate whether any solid state decomposition had occurred. The trinitroso analog of RDX (called R-salt) was identified in impacted RDX residue. Hardness testing has been used to investigate the local deformation of MgF_2 crystals (selected as a reference inert material). A considerable anistropy in hardness was found; the extent of the strain fields differed appreciably with crystal orientation. Fractoemission experiments were also performed on MgF_2 and revealed a strong crystallographic dependence for electron emission. NH_4ClO_4 exhibited a substantial hardness anisotropy in an initial study that was conducted.

FOREWORD

This work was sponsored by the Office of Naval Research under work request numbers N00014-83-WR-30046 and N00014-82-K-0263 as a cooperative effort between NSWC, White Oak, and the University of Maryland, College Park. The results and conclusions presented in this report concerning the microstructural characterization of deformed RDX explosive and a selected reference inert (MgF_2) crystal should be of interest to those studying plastic deformation and fracture in these materials. In particular, this work provides insight into their ability to concentrate energy locally as a result of being plastically deformed and fractured, leading to the initial stages of chemical decomposition. A list of references is given after the body of the report.

The authors particularly want to thank J. R. Holden for his help with the Laue x-ray diffraction experiments on RDX, and R. W. Armstrong for helpful comments and suggestions regarding this work. V. F. DeVost helped with the drop-weight impact experiments on RDX.

Approved by:

K. F. MUELLER, Head
Energetic Materials Division

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INTRODUCTION

It is well known that for initiation to occur in a solid explosive under impact conditions, the energy transferred must be concentrated into small volumes of the explosive. The most widely held view explaining this phenomenon involves the formation of "hot spots" as a result of the explosive experiencing mechanical forces. Heat is generated within a small volume at a sufficient rate to cause the temperature to rise very rapidly, the kinetics being limited by the thermal conductivity to the surrounding medium. The objective of this work is to investigate the microstructural mechanisms responsible for hot spot formation in RDX (cyclotrimethylenetrinitramine), the most common ingredient in Navy explosives, and in several reference inert materials.

In previous work,[1] surface reflection Berg-Barrett x-ray topography was used to characterize an RDX explosive single crystal, having reasonable microstructural perfection, that was grown by slow evaporation from acetone solution with seed crystals. Topographs of $(\bar{7}2\bar{1})$ and $(\bar{6}3\bar{2})$ reflections revealed a large growth strain center associated with grown-in dislocations emanating to the $(\bar{2}10)$ natural growth surface. Extensive Knoop hardness testing (50 g load) was performed on the $(\bar{2}10)$ surface in regions not influenced by the large growth strain center to assess systematically the degree of plastic anisotropy. The strain fields around the Knoop impressions were then studied using surface reflection Berg-Barrett topography ($(\bar{6}3\bar{2})$ reflection). Highly localized strain fields were observed, confirming a previous dislocation etch pit study[2] on an indented laboratory-grown crystal of apparent lesser quality.

In addition, Vickers hardness experiments (50 and 100 g load) were performed on the (001) growth face of a number of Holston production-grade Class D RDX crystals.[1,3] A considerable variation in hardness was observed and attributed to internal porosity.

A companion study involving hardness experiments and x-ray topography was performed on an MgO crystal, selected as a reference inert material.[1,4] The strain fields around spherical ball hardness impressions (1 to 100 kg load) placed into the (001) cleavage surface of the single crystal were studied using surface reflection Berg-Barrett topography ($(0\bar{2}2)$ reflection). The size of the strain fields was found to be controlled by cracking. In particular, there was a virtual absence of residual dislocations around an indentation placed at 100 kg load because they ran out {110} radial crack surfaces; this was confirmed by the inability to measure any systematic strain hardening by probing the strain fields with a Vickers indenter at low loads.

The current work further elucidates the fundamental microstructural reasons for hot spots and chemical decomposition occurring during the deformation of crystalline energetic and inert materials. The work on RDX is being primarily performed at NSWC and is closely allied with an accompanying research effort on selected model inert crystals at the University of Maryland, College Park.

X-RAY DIFFRACTION STUDIES OF RDX

Characterization of a large (several mm in size) Holston production-grade Class D RDX crystal (selected from material having NSWC designation X924) has been performed using transmission Berg-Barrett x-ray topography (Figure 1).[5] There was an absence of any appreciably enhanced diffraction intensity surrounding several pores present in the crystal (Figure 2). This result suggests that during crystallization, pore formation occurs without appreciably straining the neighboring lattice. Consequently, there is a near absence of internal strain energy available for subsequent release in these regions of the crystal. A substantial internal strain energy release is hypothesized to be necessary for the production of "hot spots."[6,7]

Using a Buerger precession camera operating in the stationary mode, transmission Laue photographs were obtained of the X924 crystal studied with x-ray topography and a crystal of comparable size selected from more recent Holston Class D material (NSWC designation X976). Both crystals exhibited the same tabular morphology reported previously by Connick and May[8] for RDX crystallized from cyclohexanone (Figure 3). However, the appearance of the diffraction spots was dramatically different (Figure 4). There was considerably more asterism for the crystal taken from X976 material, indicating that a wider spread of orientation exists in the mosaic blocks[9] (very small, slightly misoriented regions of the crystal) comprising the particular crystal. In addition, the crystal gave diffuse reflections close to some sharp reflections, a phenomenon normally attributed[9] to thermal vibration. Vickers hardness testing of the two crystals revealed a considerable difference in their hardnesses: 32-35 kgf/mm^2 for the crystal from X924 material versus 46 kgf/mm^2 for the crystal from X976 material.

Surface reflection Berg-Barrett topographic images were obtained of the strain fields around Vickers hardness impressions (50 to 450 g load) placed into a large RDX crystal that was grown from solution by Dr. H. Cady (Los Alamos National Laboratory, Los Alamos, NM). An analysis and discussion of the results will be developed in the next progress report.

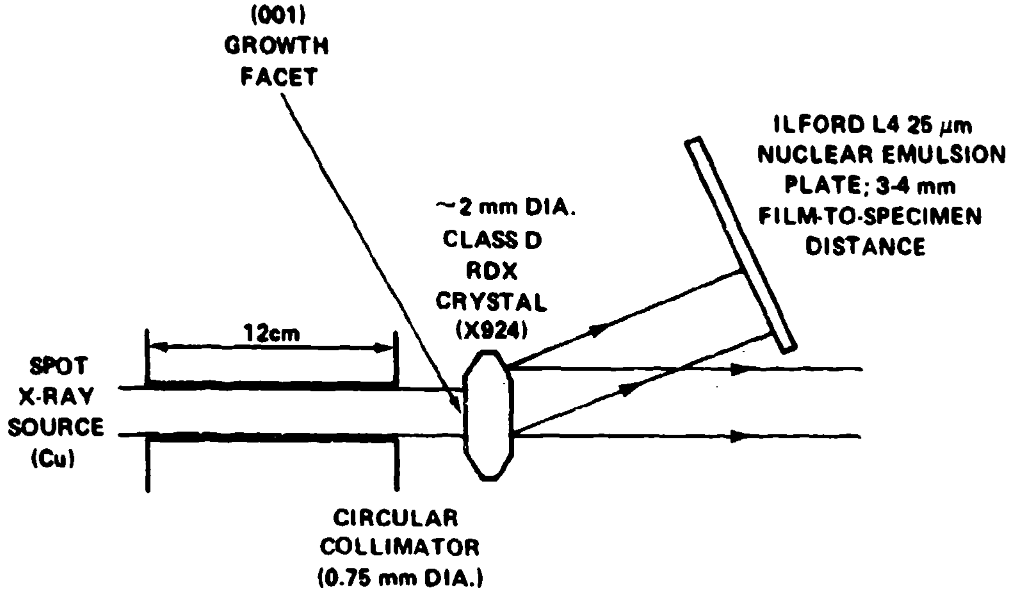
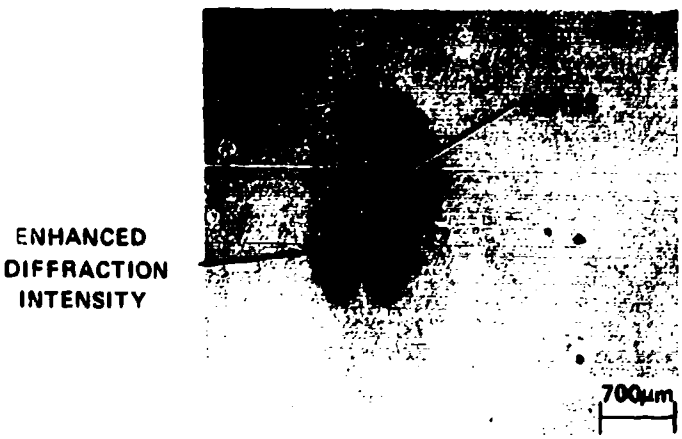
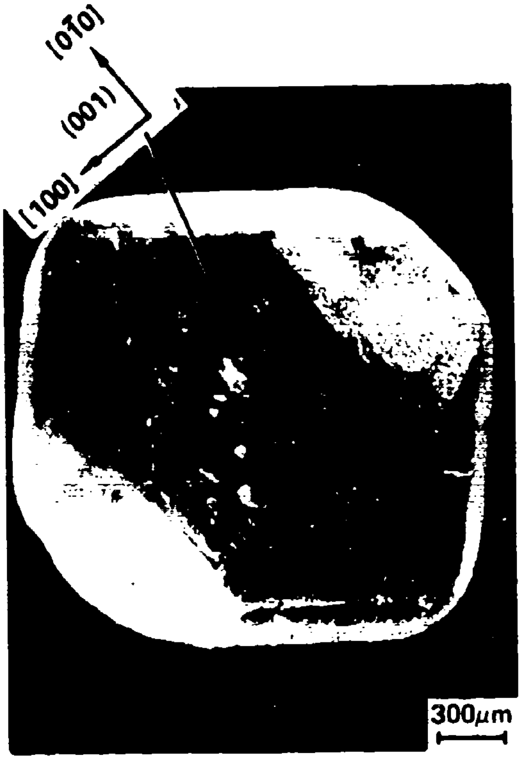


FIGURE 1. SCHEMATIC OF TRANSMISSION BERG-BARRETT X-RAY TOPOGRAPHY EXPERIMENTAL ARRANGEMENT



CuKα RADIATION AT 15 kV AND 20 mA FOR 18 MIN ON ILFORD L4 25 μm NUCLEAR PLATE

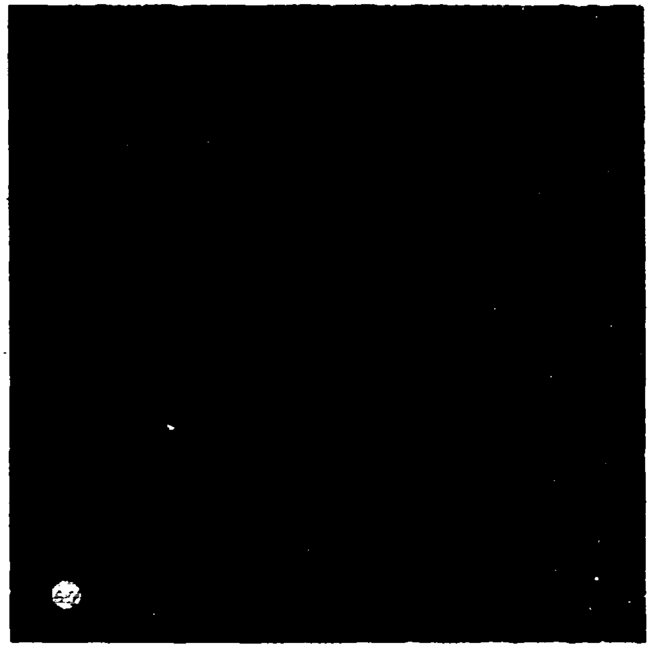
FIGURE 2. TRANSMISSION BERG-BARRETT TOPOGRAPH OF HOLSTON CLASS D RDX CRYSTAL (NSWC LOT X924)



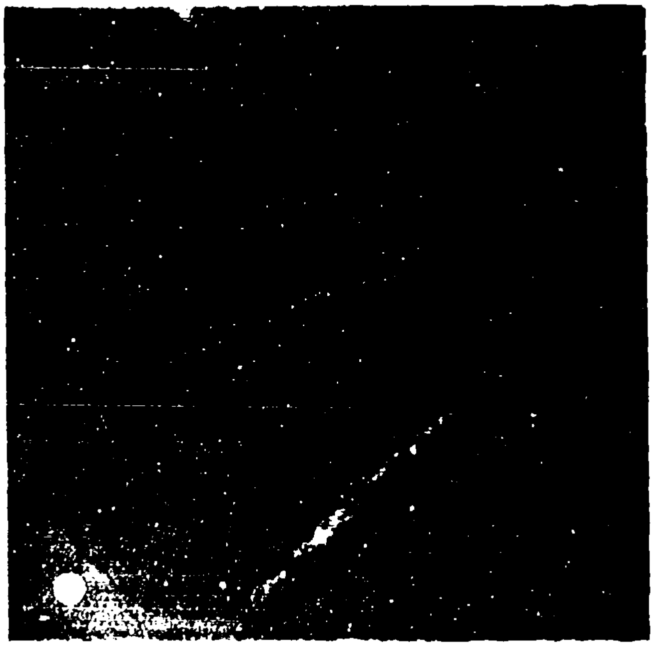
(a) NSWC LOT X924

(b) NSWC LOT X976

FIGURE 3. HOLSTON CLASS D RDX CRYSTALS



(a) NSWC LOT X924



(b) NSWC LOT X976

Mo TUBE RADIATION AT 20 kV AND 20 mA FOR
30 MIN ON ILFORD INDUSTRIAL G FILM

FIGURE 4. TRANSMISSION LAUE PHOTOGRAPHS OF HOLSTON CLASS D RDX CRYSTALS

FRACTOEMISSION EXPERIMENTS ON RDX

Fractoemission experiments[10] on production-grade Class D (X924) and NSWC laboratory-grown[2,3] RDX crystals were performed at Washington State University in collaboration with Professors J. T. Dickinson and M. H. Miles in an effort to study the very initial stages of decomposition resulting from fracture. Fracture was achieved by compressive loading (both production and laboratory crystals) and by three-point bending (laboratory crystals only). "Cleavage" type fracture resulted for several crystals subjected to three-point bending; this "singular" fracture event yielded a sharp electron emission peak followed by rapid decay (Figure 5). Multiple fracture occurred for crystals that were compressed; in this case, a large electron emission resulted and continued several minutes after fracture had occurred (Figure 6).

The fracture surfaces of some of the larger recovered fragments from five of the laboratory crystals, subjected to three-point bending, were examined by Dr. M. K. Norr (NSWC, Code R34) with the scanning electron microscope (SEM).[10] The fracture surfaces for three samples were predominantly crystallographic. The fracture surfaces for the remaining two samples examined were mostly glassy; one of these samples gave the highest electron count measured for the three-point bending experiments.

CHEMICAL DECOMPOSITION IN IMPACTED RDX

In an initial study, a series of drop-weight impact experiments (Table 1) were performed on production-grade Class D (X924) RDX crystals (0.041 g granular samples). A heat sensitive film technique[11,12] was used to detect hot spots and the generation of hot gaseous decomposition products. Gas chromatographic analyses, using a sensitive electron capture detector, were performed on the recovered, fractured samples (Figure 7(a)-(b), as examples). R-salt, the trinitroso analog of RDX (a known decomposition product of thermally degraded RDX), was conclusively identified as being formed in the production-grade RDX that had been impacted. This occurred in samples that were impacted not only at energy levels sufficient to cause the evolution of hot decomposition gases (Figure 8(a), as an example) but also at lower energy levels that resulted only in the occurrence of hot spots (Figure 8(b), as an example). However, based on the limited number of experiments performed, it is unclear how the nitroso compound was formed.

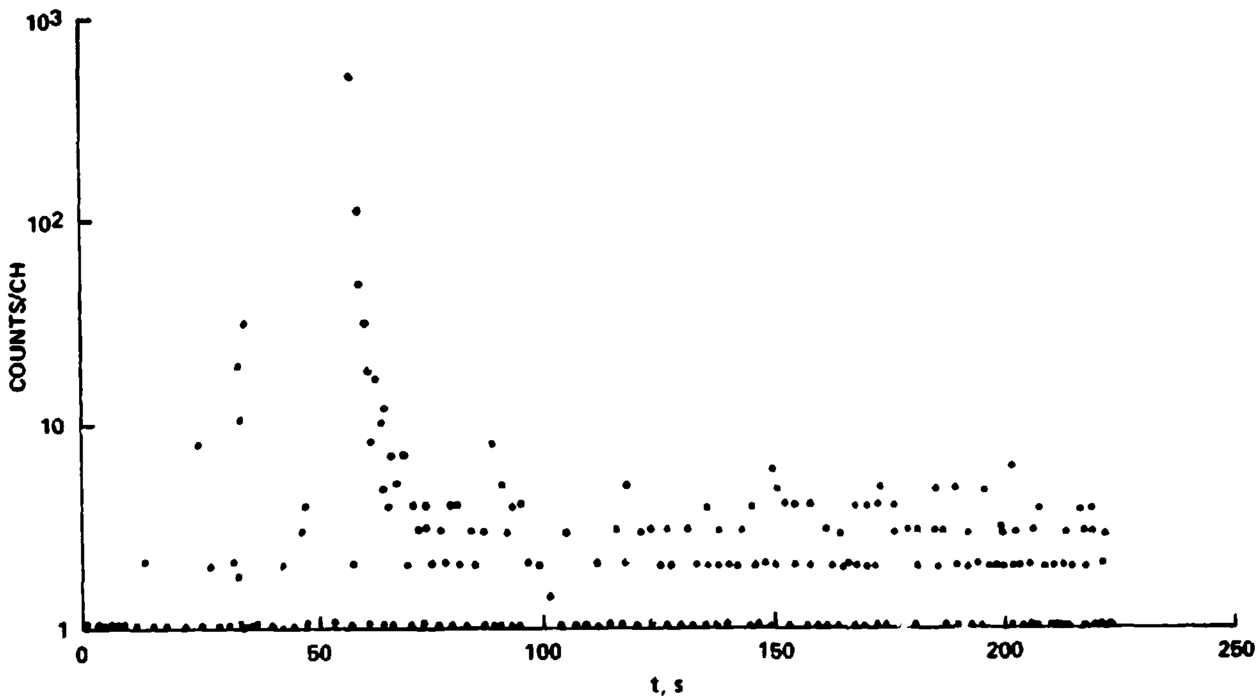


FIGURE 5. ELECTRON EMISSION FROM NSWC LABORATORY-GROWN RDX CRYSTAL FRACTURED BY THREE-POINT BENDING (AFTER REFERENCE 10)

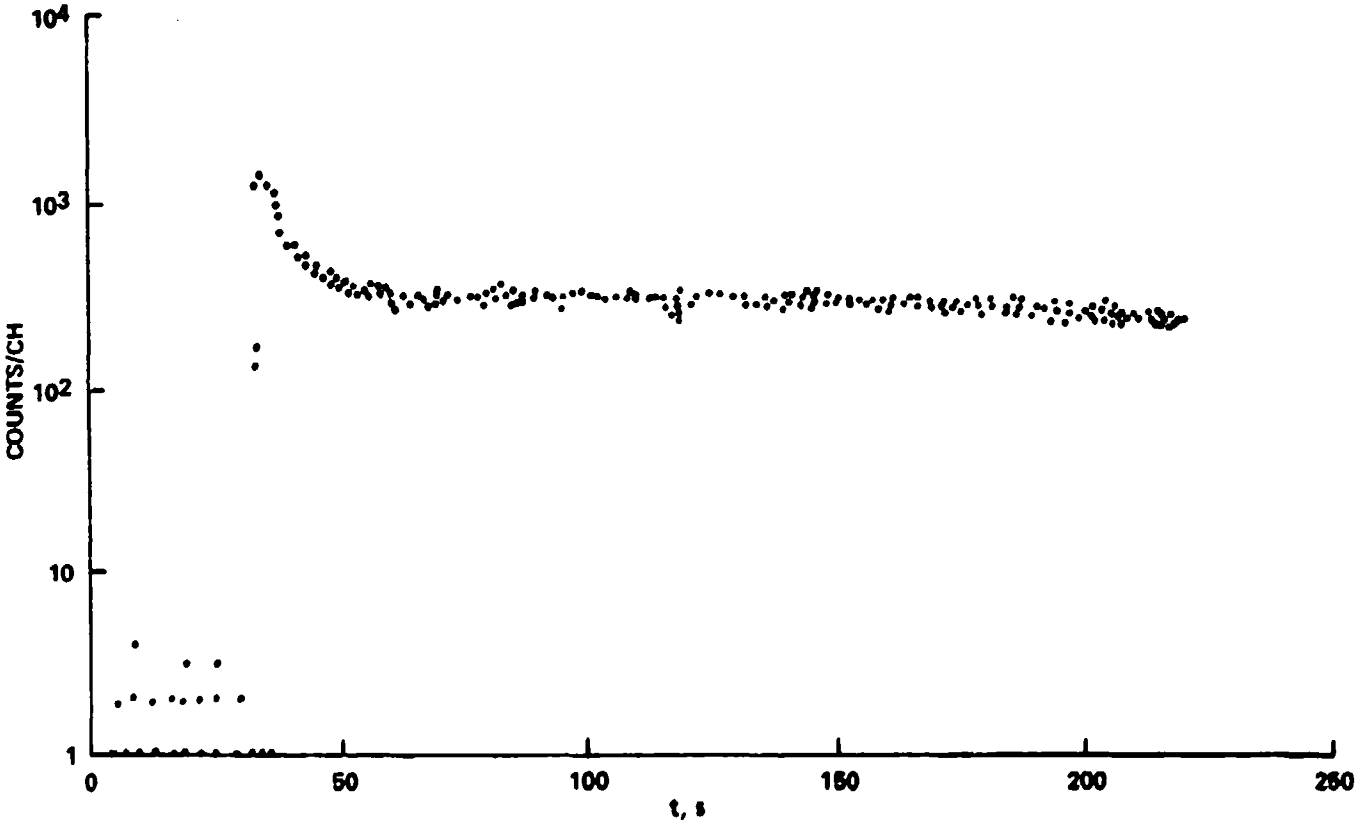


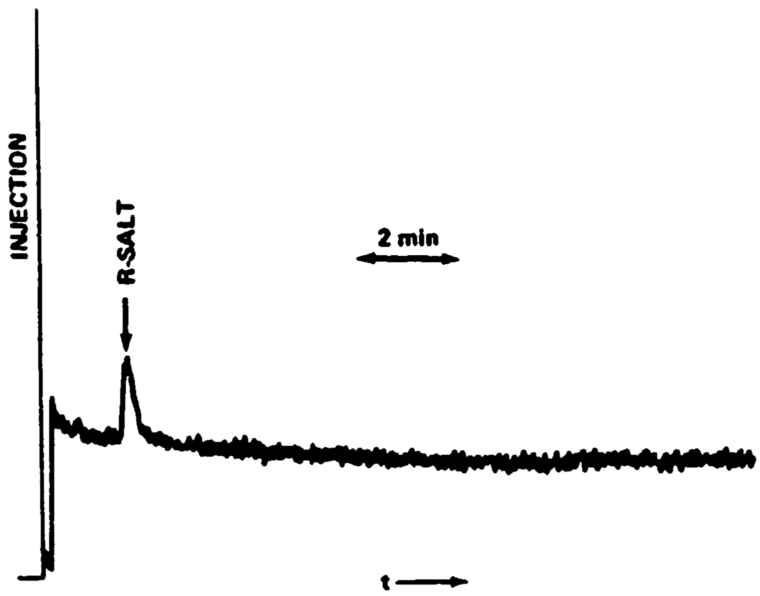
FIGURE 6. ELECTRON EMISSION FROM NSWC LABORATORY-GROWN RDX CRYSTAL FRACTURED IN COMPRESSION (AFTER REFERENCE 10)

TABLE 1. SUMMARY OF DROP-WEIGHT IMPACT EXPERIMENTS ON CLASS D RDX (NSWC LOT X924)

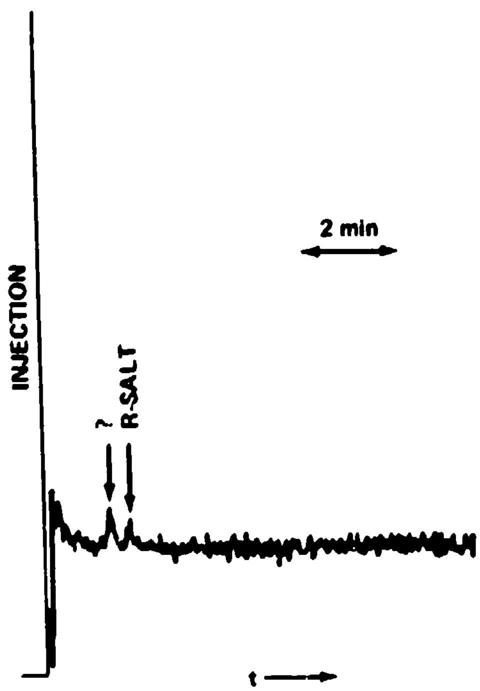
(0.041 g Sample: Loose Pile)

Experiment Number	Drop* Height (cm)	Go/No Go	Comments
1	10	Go**	---
2	5	No Go	Tiny Amount of Browning at Center***
3	8	No Go	Tiny Amount of Browning at Center***
4	9	No Go	Tiny Amount of Browning at Center***
5	9.5	No Go	Tiny Amount of Browning at Center***
6	9.8	Go**	---
7	9.7	No Go	Considerable Amount of Browning at Center***

*5 kg mass
**As indicated by browning of film beyond the extent of the impacted sample (Figure 8(a))
***Visual observation of heat sensitive film discoloration

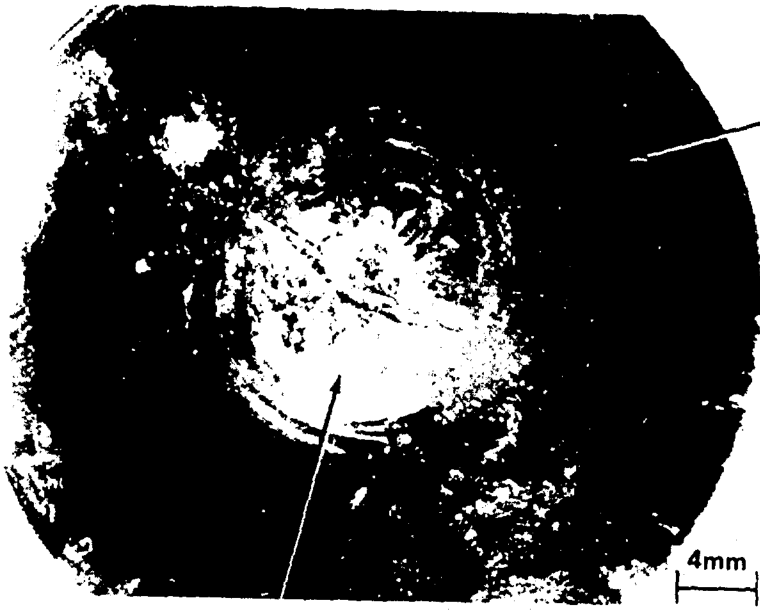


(a) EXPERIMENT #1 (10 cm DROP HEIGHT: GO)

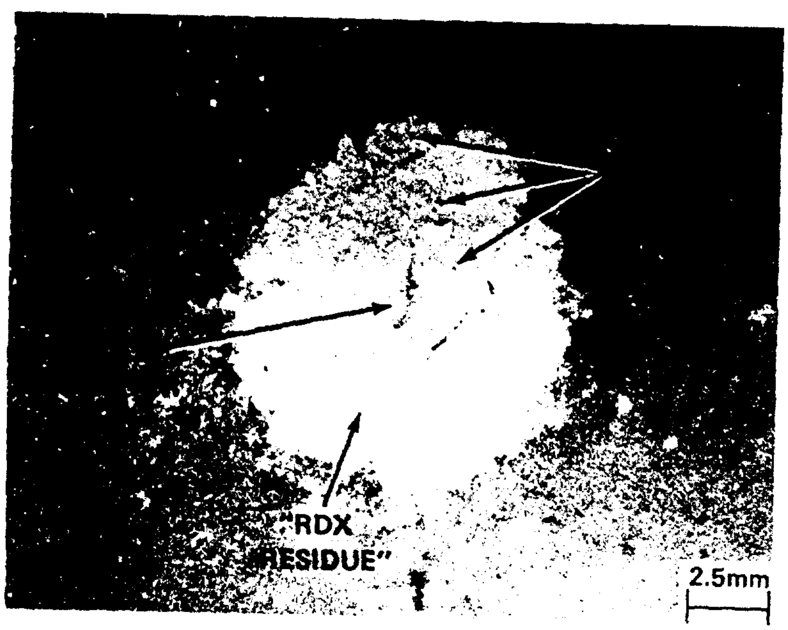


(b) EXPERIMENT #2 (5 cm DROP HEIGHT: NO GO)

FIGURE 7. GAS CHROMATOGRAPHIC ANALYSES OF RECOVERED HOLSTON CLASS D RDX (NSWC LOT X924) THAT WAS DROP-WEIGHT IMPACTED



(a) EXPERIMENT #1 (10 cm DROP HEIGHT: GO)



(b) EXPERIMENT #2 (5 cm DROP HEIGHT: NO GO)

FIGURE 8. HEAT SENSITIVE FILM RECORDS OF DROP-WEIGHT IMPACTED HOLSTON CLASS D RDX (NSWC LOT X924)

EXPERIMENTAL INVESTIGATION OF HOT SPOT FORMATION DURING IMPACT

In a separate study, drop-weight impact experiments were performed on pressed samples (0.035 g) of PETN (pentaerythritoltetranitrate) and Comp A-3 (RDX/wax = 91/9) using the well instrumented[13] NSWC impact machine. The heat generated on impact was measured by a fast, broadband (1-13 μm) infrared detector. Initial, low level stages ($\Delta T \approx 200°C$) of chemical reaction were observed for both materials while being impacted. However, to date it has not been possible to detect heating prior to the initial stages of chemical reaction. This may be a matter of detector sensitivity, and the problem is being addressed.

LOCAL PLASTIC DEFORMATION IN MgF_2

The nature of the plastic deformation resulting from performing hardness experiments on single crystals of MgF_2 (supplied by Harshaw) was investigated at the University of Maryland, College Park, using light microscopy and surface reflection Berg-Barrett x-ray topography.[5,14,15]

Optical examination of hardness impressions put into the (110), (100), (101), (001), and (111) surfaces of MgF_2 using a 1.59 mm (0.0625 in.) diameter spherical indenter (10 kg load) revealed very different deformation features. However, it was determined that these hardness impressions were all reproducible. As examples, a hardness impression on the (110) and (111) surfaces appears in Figures 9 and 10, respectively, together with an appropriate stereographic projection providing angular information for certain directions (open symbol) and plane normals (closed symbol).

Knoop hardness measurements (50 g load) were obtained for different directions in the (110), (001), (101), (100), and (111) crystal surfaces of MgF_2. The Knoop hardness indentations put into the (110) surface appear at low magnification in Figure 11. The results for all of the surfaces appear in Table 2 and show that a considerable hardness anisotropy exists for each surface; to this extent, MgF_2 should serve as an appropriate model material for RDX since it also exhibited[1-3] a significant hardness anisotropy. With one exception, no cracking occurred for those indentations in which the long axis of the indenter was aligned parallel to $[1\bar{1}5]$, which was also the hard direction for this surface; hardness impressions for the other directions had cracks surrounding them. This result also relates to previous results[1,2] obtained on RDX crystals as well.

The residual strain fields surrounding various hardness indentations in MgF_2 were investigated using surface reflection Berg-Barrett topography. Figure 12 is an (040) reflection topograph of the area shown optically in Figure 11; the topographic strain fields appear as black regions of enhanced diffracted intensity. The strain fields for [001] impressions were reasonably circular while the strain fields for the other impressions were anisotropic. Higher

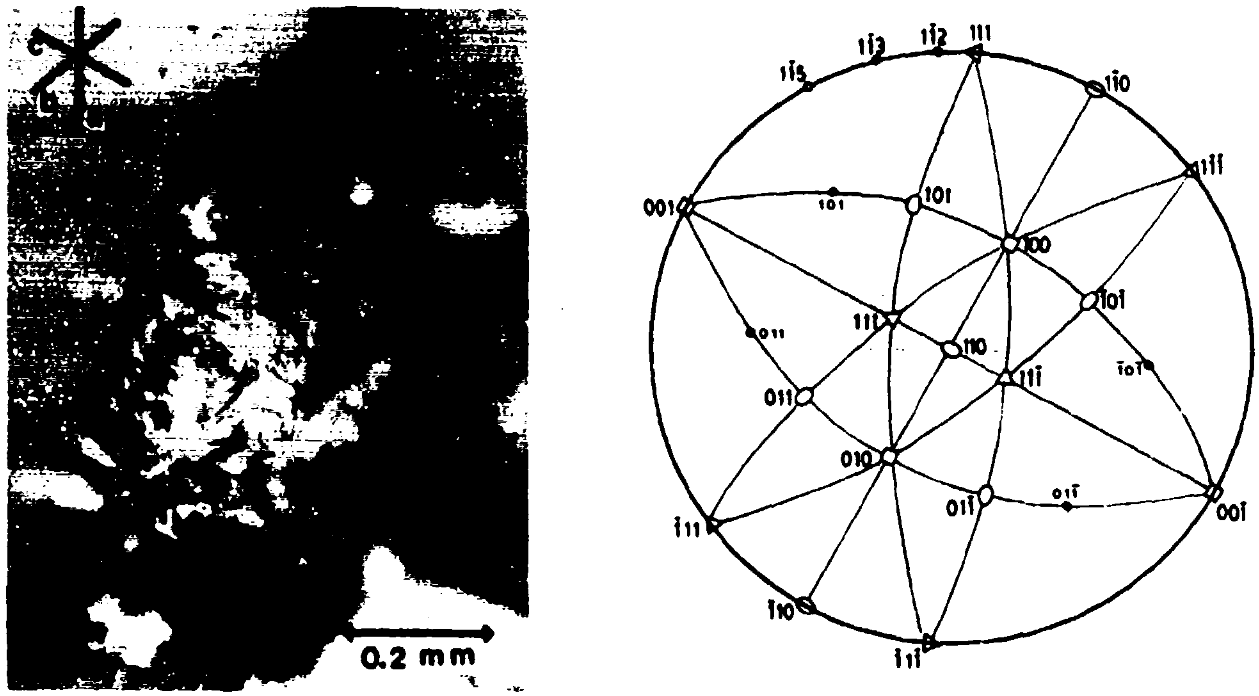


FIGURE 9. HARDNESS IMPRESSION (SPHERICAL INDENTER) IN THE (110) SURFACE OF MgF_2; AND, STEREOGRAPHIC PROJECTION DESCRIPTION

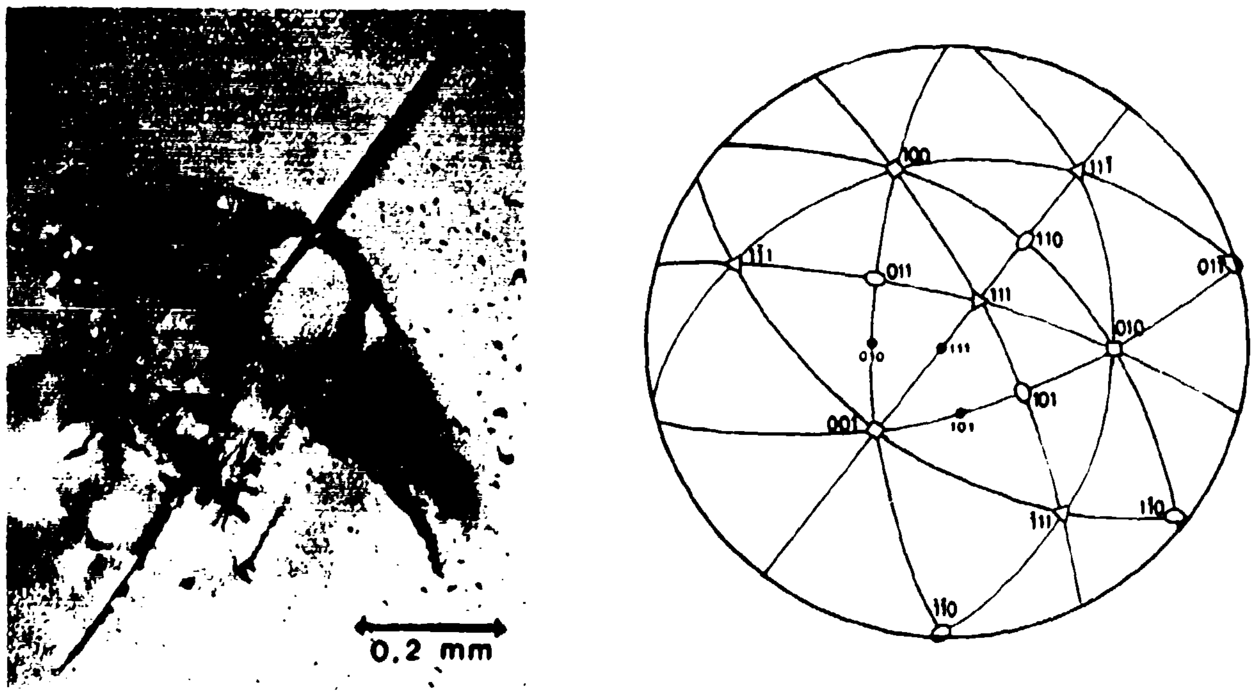


FIGURE 10. HARDNESS IMPRESSION (SPHERICAL INDENTER) IN THE (111) SURFACE OF MgF_2; AND, STEREOGRAPHIC PROJECTION DESCRIPTION

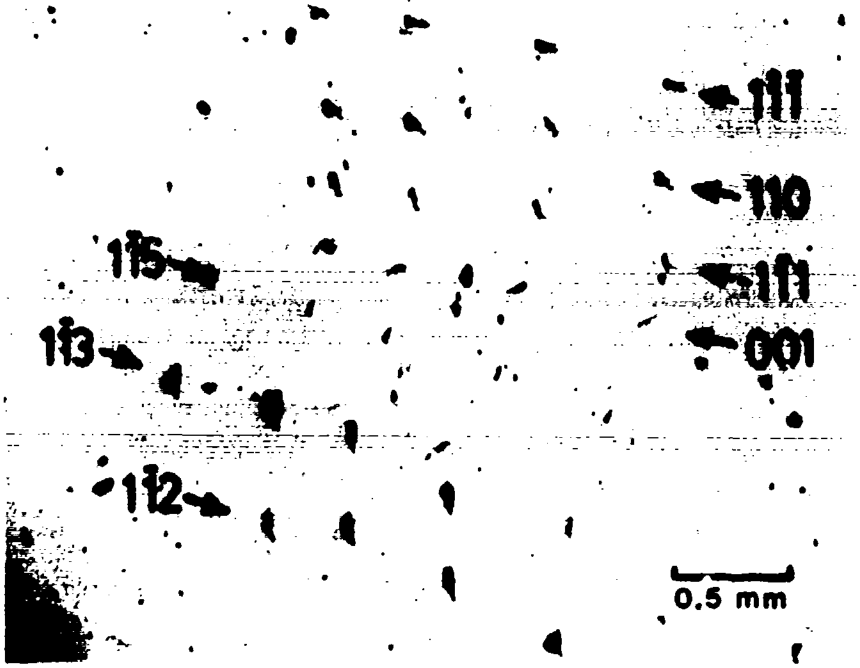
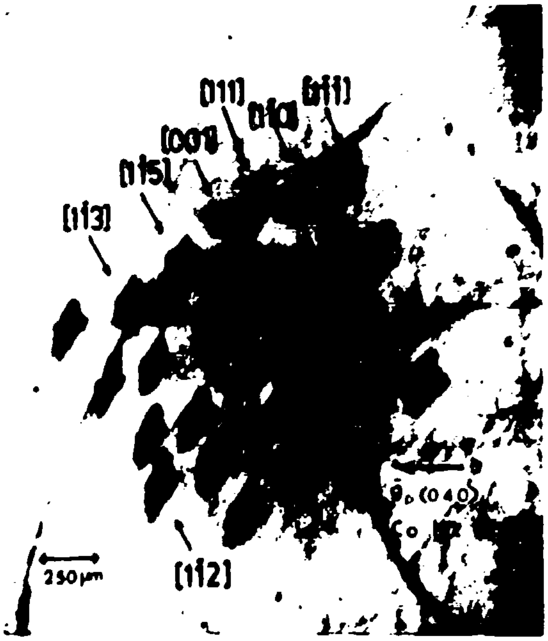


FIGURE 11. KNOOP HARDNESS IMPRESSIONS IN THE (110) SURFACE OF MgF_2



RADIATION AT 30 kV AND 10 mA FOR 3 h
ON ILFORD L4 50 μm NUCLEAR PLATE

FIGURE 12. SURFACE REFLECTION BERG-BARRETT TOPOGRAPH OF KNOOP HARDNESS IMPRESSIONS IN THE (110) SURFACE OF MgF_2

TABLE 2. KNOOP HARDNESS VALUES OBTAINED FOR VARIOUS MgF_2 CRYSTAL SURFACES

(50 g Load)

Surface	Hardness (kgf/mm^2)	Orientation of Indenter Axis
(110)	268	[001]
	458	$[1\bar{1}5]$
	232	$[1\bar{1}3]$
	139	$[1\bar{1}2]$
	280	[111]
	214	$[1\bar{1}0]$
	242	$[1\bar{1}\bar{1}]$
(001)	122	[010]
	455	[100]
(101)	444	$[0\bar{1}0]$
	94.4	$[1\bar{1}1]$
	163	$[10\bar{1}]$
(100)	211	[001]
	133	[011]
	270	[010]
	416	$[01\bar{1}]$
(111)	310	$[\bar{1}01]$
	128	[011]
	94.5	$[1\bar{1}0]$

magnification topographs of the hardness impressions placed in the (110) and (111) surfaces using a spherical indenter are shown in Figures 13 and 14, respectively. These two topographs also show a contrast in the degree of anisotropy of the strain fields. In addition, the extent of the strain field for each impression differs appreciably. The strain fields around hardness impressions in MgF_2 are not similar in this respect to those observed in RDX, where there was a virtual absence of enhanced diffracted intensity[1] which is consistant with the findings from etch pit studies.[2,16]

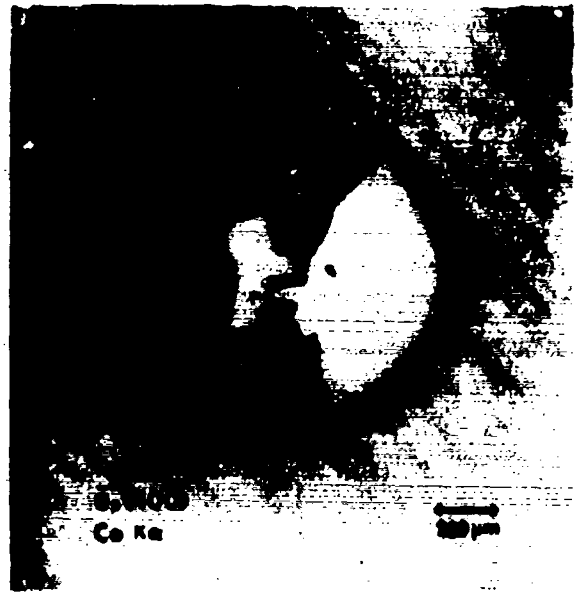
FRACTOEMISSION EXPERIMENTS ON MgF_2

Fractoemission experiments on single crystals of MgF_2 (supplied by Harshaw) were performed at Washington State University in a collaborative effort between Professor J. T. Dickinson and researchers at the University of Maryland, College Park. The specimen dimensions were 10 mm x 5 mm x 1 mm thick. Fracture was achieved by three-point bending using a knife edge; the knife axis was applied normal to either the (110) or (101) surfaces. This resulted in either the (101) or (110) fracture surfaces, respectively. The electron emission results are given in Table 3 for a number of samples and show a marked dependence on the crystallographic orientation. The fracture surfaces were examined in the SEM. The (101) fracture surface was found to consist of two {101} planes while only a single, flat (110) fracture surface resulted. This behavior is opposite to that observed for RDX where a "simple" fracture gave a lower total emission.[10]

TABLE 3. ELECTRON EMISSION FROM MgF_2 CRYSTALS FRACTURED BY THREE-POINT BENDING

Fracture Surface	Electron Emission (Total Count)			
	Sample #1	#2	#3	#4
(101)	5,500	6,700	5,000	10,000
(110)	433,000*	22,900	22,500	34,700

*A part of the fractured sample was on the detector.



RADIATION AT 30 kV AND 10 mA FOR 3 h ON ILFORD L4 50 μm NUCLEAR PLATE

FIGURE 13. SURFACE REFLECTION BERG-BARRETT TOPOGRAPH OF HARDNESS IMPRESSION (SPHERICAL INDENTER) IN THE (110) SURFACE OF MgF_2

RADIATION AT 30 kV AND 10 mA FOR 3 h ON ILFORD L4 50 μm NUCLEAR PLATE

FIGURE 14. SURFACE REFLECTION BERG-BARRETT TOPOGRAPH OF HARDNESS IMPRESSION (SPHERICAL INDENTER) IN THE (111) SURFACE OF MgF_2

PLASTIC ANISOTROPY IN NH_4ClO_4

In an initial attempt to assess the plastic anisotropy of ammonium perchlorate (NH_4ClO_4), Knoop hardness testing (50 g load) was performed on the (001) growth surface of a high quality, pure single crystal (Apache: Code No. 1-3-20-0) supplied by T. Boggs (Naval Weapons Center, China Lake, CA). The hardness anisotropy for this surface is shown in Figure 15, with a minimum hardness being measured for the $[1\bar{1}0]$ direction. The hardness was highest for $[\bar{1}00]$ and [010] directions. Although this hardness anisotropy is substantial, it is not as pronounced as has been obtained previously for RDX.[1-3]

SUMMARY

Through-transmission Berg-Barrett and Laue x-ray diffraction techniques have been used to make significant microstructural observations about the state of growth perfection in Class D (Holston) production-grade RDX crystals. It was determined that pore formation has occurred in these crystals without appreciably straining the surrounding lattice structure. It was also possible to detect a variation in the spread of orientation for mosaic blocks in two lots of crystals. In collaboration with researchers at Washington State University, fractoemission experiments were performed on production-grade and laboratory-grown RDX crystals to investigate the events that take place in the initial stages of decomposition in fractured crystals. Work commenced to identify chemically any solid state decomposition products in impacted production-grade RDX crystals at energy levels below and above that required to cause gaseous decomposition. The trinitroso analog of RDX, R-salt, was successfully identified by gas chromatographic analyses of impacted "RDX residues". The combined work aids our understanding, on a microstructural level, into how "hot spots" can form in crystalline explosives. Further, knowledge of the sub-initiation events that occur in fractured RDX crystals is being acquired.

A companion study involving hardness experiments and Berg-Barrett topography (to assess the strain fields surrounding hardness impressions) was performed on MgF_2. This material was selected as a model inert, for one reason, because it has a non-cubic unit cell. A considerable hardness anisotropy was measured for MgF_2 as was the case for RDX. The significant observation was made that the strain fields around hardness impressions in MgF_2 were not localized as in RDX. Fractoemission experiments were also performed on MgF_2 at Washington State University; electron emission was found to have a strong crystallographic dependence. An initial study of the plastic anisotropy of NH_4ClO_4 was performed; a substantial hardness anisotropy was measured, although not as great as for RDX.

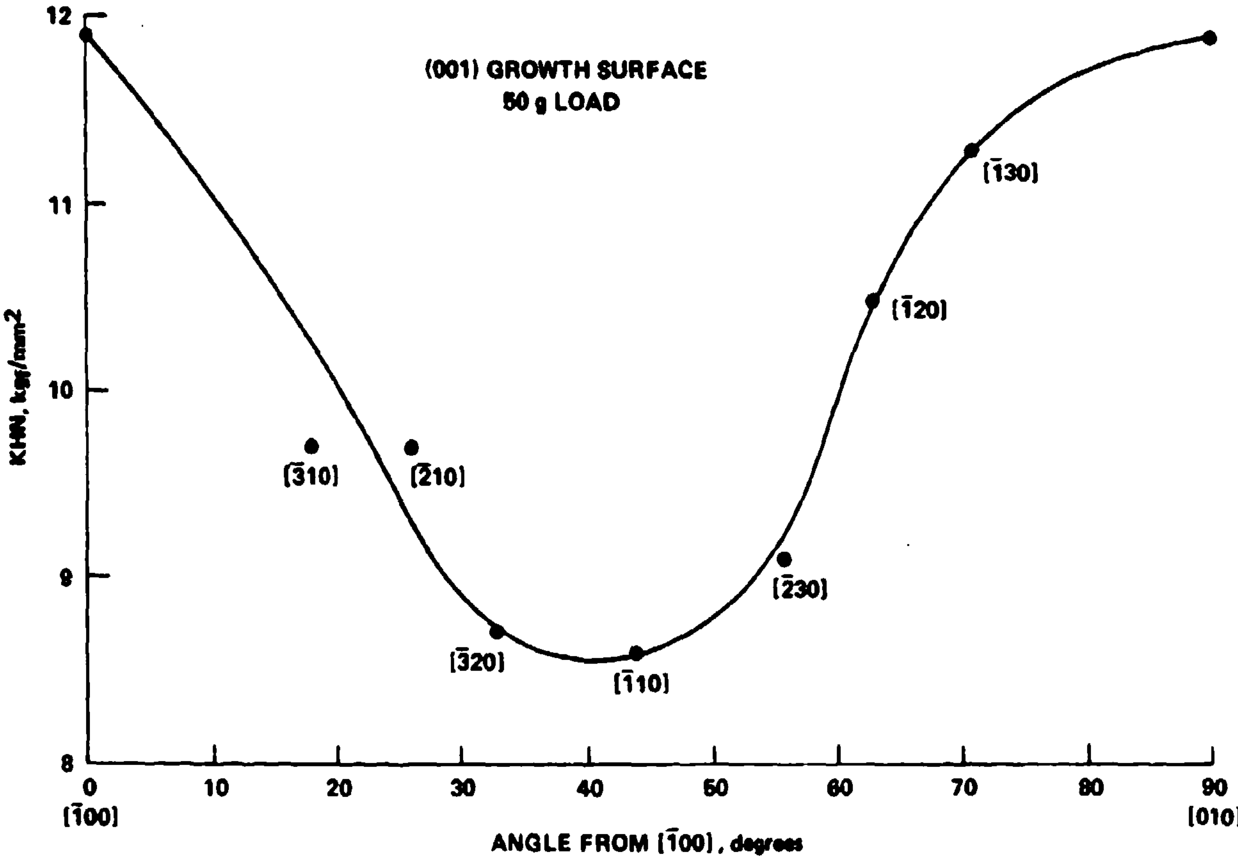


FIGURE 15. KNOOP HARDNESS ANISOTROPY FOR LABORATORY-GROWN NH_4ClO_4 CRYSTAL

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